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Method of Crystallizing Polyester

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SPECIFICATION

1. Title of the Invention

Method of Crystallizing Polyester

2. Claims

1. A method of crystallizing polyester, characterized in that polyester powder whose primary repeating units are alkylene terephthalate is crystallized by being treated with steam heated to 110°C or higher.

3. Detailed Description of the Invention

The present invention relates to a method of crystallizing polyester powder.

The purpose of the present invention is to provide an improved method of crystallizing polyester powder.

When polyester is directly melted while still moist, the ester bonds hydrolyze and the degree of polymerization decreases, becoming a source of a considerable reduction in the quality of the product obtained by means of spinning or molding. Therefore, when polyester is melt-spun or melt-molded, this polyester is usually thoroughly heated in powder form prior to melting in order to eliminate the moisture content as much as possible. Moreover, the solid-phase polymerization method whereby polyester powder with a relatively low degree of polymerization is heated to raise the degree of polymerization is also widely employed.

Nevertheless, when the polyester powder is directly submitted to heating and drying or to solid-phase polymerization in these cases, aggregation of the particles occurs during heating, making trouble-free heating impossible. Therefore, a method is proposed whereby the polyester that is to be submitted to drying or solid-phase polymerization is pre-heated to the crystallization temperature or higher and then dried or polymerized in the solid phase.

Examples of conventional crystallization methods of this type include a method whereby polyester powder is placed on a fluid bed and crystallized as hot air flows through the powder, a method of crystallization by means of hot air in a rotary dryer, a method of crystallization by means of hot air while agitating in a crystallization tank with agitating blades, and the like.

However, there are problems with these methods of crystallization by means of hot air in that it is impossible to prevent aggregation of the powder particles during the crystallization process, and if crystallization treatment is not performed for a long time of 20 minutes or more, thorough crystallization does not occur, so energy costs are very high.

On the other hand, a method has also been proposed whereby crystallization treatment is performed with water or steam at 80 to 100°C (USP 836,742). However, problems occur with this method in that it takes a long time, 15 to 25 minutes, to crystallize polyester powder, and

because moist powder enters the drying and solid-phase polymerization zones, hydrolysis occurs during drying and solid-phase polymerization.

The inventors performed intense studies to solve these problems of prior art, and, as a result, they completed the present invention upon discovering that when crystallization treatment is performed using steam heated to 110°C or higher, the powder can be crystallized in a very short time, and the moisture content will not enter the drying or solid-phase polymerization zones.

That is, the present invention is a method of crystallizing polyester characterized in that polyester powder whose primary repeating units are alkylene terephthalate is crystallized by being treated with steam heated to 110°C or higher.

The polyester in the present invention is one whose primary repeating units are alkylene terephthalate, and polyester whose primary repeating units are ethylene terephthalate is particularly preferred. A third component may also be copolymerized at 10 mol% or less. Examples of such components include dibasic acids such as adipic acid, sebacic acid, isophthalic acid, 5-sodium sulfoisophthalate, and naphthalene dicarboxylic acid; oxy acids such as oxybenzoic acid; and glycols such as diethylene glycol, propylene glycol, neopentyl glycol, pentaerythritol, and polyethylene glycol. Another polymer may also be blended at 10 wt% or less. Moreover, delustering agents (such as titanium oxide), flame retardants, weathering agents, heat resistors, coloration stabilizers (such as phosphorus compounds), antistatic agents, fluorescent brighteners, and viscosity stabilizers (such as boron compounds) can be added to this polyester.

The polyester powder in the present invention means polyester in chip, pellet, flake, or powder form.

The steam that is used in the present invention must be steam that has been heated to 110°C or higher. Crystallization in a short amount of time is impossible when the temperature is lower than 110°C. Either saturated steam or superheated steam can be used as the hot steam. However, when saturated steam is used, there is the chance that moisture will remain in the

powder and hydrolysis will occur during the drying and solid-phase polymerization processes, so superheated steam is preferred.

A treatment time of 1 to 2 minutes with steam heated to 110°C, 10 seconds to 1 minute with steam heated to 130°, or 5 to 20 seconds with steam heated to 150°C is sufficient. Crystallization can be accomplished in a very short time when contrasted to the fact that crystallization with hot air requires a treatment time of 20 minutes or longer.

Thus, by means of the present invention, polyester powder can be crystallized in a very short time of no more than one-tenth the time of the prior art, and there is almost no aggregation of particles or no detrimental effect from the moisture content during the drying and solid-phase polymerization processes. Therefore, the present invention makes a very large contribution to reducing energy costs and improving treatment results.

The present invention will now be described in further detail with working examples.

Working Examples 1 through 3

Polyethylene terephthalate with a melting point of 260°C and a limiting viscosity [η] of 0.64, obtained by means of a melt polymerization of dimethyl terephthalate and ethylene glycol, was extruded, quenched, and cut into cylindrical pellets with a diameter of 3 mm and length of 4 mm. These pellets were placed on a metal screen and crystallized by means of blowing steam heated to various temperatures from below the screen through the pellets. Next, the pellets were introduced to a vacuum rotary dryer shaped as a double cone and heated to 180°C. The pellets were heated and dried under reduced pressure as the temperature was raised from 180°C to 230°C over a period of 3 hours, and the dried pellets were polymerized in the solid phase. In this case, the treatment time with superheated steam was varied, and it was determined how long the pellets polymerized in the solid phase needed to be treated with hot steam before aggregation stopped. The results are shown in the following table.

	Hot steam temperature (°C)	Treatment time (minutes)
Example 1	110	1
Example 2	130	0.2
Example 3	150	0.1

No aggregation at all was seen with the pellets after treatment with hot steam, and there were no problems associated with the moisture content during the drying or solid-phase polymerization processes.

Comparative Examples 1 and 2

By way of comparison, crystallization treatment was performed using steam heated to 150°C and saturated steam at 100°C in place of the steam heated to 110°C in Working Example 1. The experiment was repeated with the other conditions being the same as in Working Example 1, and it was determined how long the pellets polymerized in the solid phase had needed to be treated with hot steam before aggregation stopped. The results are shown in the following table.

	Crystallization treatment conditions	Treatment time (minutes)	Comments
Comparative Example 1	Hot air at 150°C	20	Some aggregation occurred after crystallization treatment
Comparative Example 2	Saturated steam at 100°C	15	Hydrolysis was caused by the moisture content during the heating and drying process

As is clear from these results, when crystallization treatment is performed using steam heated to 110°C, polyester powder can be crystallized in a very short time with no aggregation of the pellets during crystallization treatment or problems associated with the moisture content in the drying and solid-phase polymerization process.

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